FOOD CHEMICALS CODEX 3RD ED
FIRST AND SECOND SUPPLEMENT 93 96

FIRST SUPPLEMENT TO THE THIRD EDITION

FOOD CHEMICALS CODEX

COMMITTEE ON FOOD CHEMICALS CODEX

Food and Nutrition Board
Commission on Life Sciences
National Research Council

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To the memory of

JUSTIN L. POWERS, Ph.D.

1895-1981

First Director of the *Food Chemicals Codex,* 1961-66

Organization of the *Food Chemicals Codex* (1981–83)

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Contents

Additions, Changes, and Corrections

Additions, changes, and corrections listed herein constitute revisions in the *Food Chemicals Codex*, Third Edition (FCC III). Page numbers refer to FCC III unless indicated by a reference to pages in THIS SUPPLEMENT.

1/ *General Provisions Applying to Specifications, Tests, and Assays of the* Food Chemicals Codex

No Change.

2/ *Monographs*

Insert the following new monograph to precede the monograph entitled *Adipic Acid*, page 11:

Acid Hydrolyzed Proteins

Hydrolyzed Vegetable Protein (HVP); Hydrolyzed Plant Protein (HPP); Hydrolyzed Milk Protein

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DESCRIPTION

Acid hydrolyzed proteins are composed primarily of amino acids and salts resulting from the acid-catalyzed breakdown of peptide bonds present in edible proteinaceous materials. In processing, the protein hydrolysates may be treated with safe and suitable alkaline materials. The edible proteinaceous materials used as raw materials are derived from corn, soy, wheat, yeast, peanut, rice, or other safe and suitable vegetable sources, or from milk. Individual products may be in liquid, paste, powder, or granular form. The pH of a 2% solution in water is between 4.3 and 7.0.

REQUIREMENTS

Calculate all analyses on the dried basis. Liquid and paste samples should be evaporated to dryness on a steam bath, then, as for the powdered and granular forms, dried to constant weight at 105° (see *General Provisions Applying to Specifications, Tests, and Assays of the Food Chemicals Codex*, page 1).

Assay (Total Nitrogen) Not less than 3.25% total nitrogen.
α-Amino Nitrogen Not less than 2.0%.
Arsenic Not more than 3 ppm.
Aspartic Acid Not more than 6.0% as $C_4H_7NO_4$ and not more than 15.0% of the total amino acids.
Glutamic Acid Not more than 20.0% as $C_5H_9NO_4$ and not more than 35.0% of the total amino acids.
Heavy Metals (as Pb) Not more than 0.002%.
Insoluble Matter Not more than 1%.
Lead Not more than 10 ppm.
Sodium Not more than 25.0%.

TESTS

Assay (Total Nitrogen) Proceed as directed under *Nitrogen Determination*, page 521.

α-Amino Nitrogen Transfer 7 to 25 g, accurately weighed, into a 500-ml volumetric flask with the aid of several 50-ml portions of warm ammonia-free water, dilute to volume with water, and mix. Neutralize 20.0 ml of the solution with 0.2 *N* barium hydroxide or 0.2 *N* sodium hydroxide, using phenolphthalein TS as indicator, and add 10 ml of freshly prepared phenolphthalein-formol solution (50 ml of 40% formic acid containing 1 ml of 0.05% phenolphthalein in 50% alcohol neutralized exactly to pH 7 with 0.2 *N* barium hydroxide or sodium hydroxide). Titrate with 0.2 *N* barium hydroxide to a distinct red color, add a small but accurately measured volume of 0.2 *N* barium hydroxide in excess, and back titrate to neutrality with 0.2 *N* hydrochloric acid. Conduct a blank titration using the same reagents, with 20 ml of water in place of the test solution. Each ml of 0.2 *N* barium hydroxide is equivalent to 2.8 mg of α-amino nitrogen.

Arsenic A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Arsenic Test*, page 464.

Aspartic and Glutamic Acids

Apparatus Use an ion-exchange amino acid analyzer, equipped with sulfonated polystyrene columns, in which the effluent from the sample is mixed with ninhydrin reagent and the absorbance of the resultant color is measured continuously and automatically at 570 and 440 nm by a recording photometer.

Standard Solution Prepare a standard mixture of amino acids as follows: Weigh 1250 ± 2 μmol of each amino acid, and place in a 500-ml volumetric flask. Fill the flask half-full with water, and add 5 ml of concentrated hydrochloric acid to dissolve the less-soluble amino acids. Prepare the standard for analysis by diluting 1 ml of this solution with 4 ml of 0.2 *N* sodium citrate, pH 2.2, buffer. Each 2-ml aliquot contains 1.00 μmol of each amino acid.

Sample Preparation Accurately weigh 5 mg of the sample, and dilute to exactly 5 ml with 0.2 *N* sodium citrate, pH 2.2, buffer. Remove any insoluble material by centrifugation or filtration.

Procedure Using 2-ml aliquots of the *Standard Solution* and *Sample Preparation*, proceed as directed according to the apparatus manufacturer's instructions. From the chromatograms thus obtained, match the retention times produced by the *Standard Solution* with those produced by the *Sample Solution*, and identify the peaks produced by aspartic acid and glutamic acid. Record the area of the respective amino acid peak from the sample as A_A, and that from the standards as A_S.

Calculations Calculate the respective concentration, C_A, in μmol per ml, of aspartic acid and glutamic acid in the *Sample Preparation* by the formula $A_A \times C_S / A_S$, in which C_S is the concentration, in μmol per ml, of the respective amino acid in the *Standard Solution*.

Calculate the respective percentage of aspartic acid and glutamic acid, on the basis of total amino acids, by the formula $16 \times C_A/N_T$, in which N_T is the percentage of total nitrogen determined in the *Assay*.

Calculate the respective percentage of aspartic acid and glutamic acid in the sample by the formula $100 \times C_A/S_W$, in which S_W is the weight of the sample taken, in g.

Heavy Metals Prepare and test a 1-g sample as directed in *Method II* under the *Heavy Metals Test*, page 513, using 20 μg of lead ion (Pb) in the control (*Solution A*).

Insoluble Matter Transfer about 5 g, accurately weighed, into a 250-ml Erlenmeyer flask, add 75 ml of water, cover the flask with a watch glass, and boil gently for 2 min. Filter the solution through a tared filtering crucible, dry at 105° for 1 h, cool, and weigh.

Lead A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Lead Limit Test*, page 518, using 10 μg of lead ion (Pb) in the control.

Sodium

Spectrophotometer Use any suitable atomic absorption spectrophotometer.

Standard Solution Transfer 25.42 mg of reagent-grade sodium chloride, accurately weighed, into a 1000-ml volumetric flask, dissolve in and dilute to volume with deionized water, and mix. Transfer 5.0 ml of this solution to a second 1000-ml volumetric flask, dilute to volume with deionized water, and mix. Each ml contains 0.5 μg of Na.

Sample Solution Transfer 1.00 ± 0.05 g of previously dried sample, accurately weighed, into a silica or porcelain dish. Ash in a muffle furnace at 246°–260° for 2–4 h. Allow the ash to cool, and dissolve in 5 ml of 20% HCl, warming the solution if necessary to complete solution of the residue. Filter the solution through acid-washed filter paper into a 500-ml volumetric flask. Wash the filter paper with hot water, dilute to volume, and mix.

Procedure Determine the absorbance of each solution at 589.0 nm, following the manufacturer's instructions for optimum operation of the spectrophotometer. The absorbance produced by the *Sample Solution* does not exceed that of the *Standard Solution*.

Packaging and Storage Store in well-closed containers.
Functional Use in Foods Flavoring agent.

Aluminum Ammonium Sulfate, page 14

Replace the *Test* entitled *Assay*, page 15, with the following (note that the buffer is added prior to the boiling step):

Assay Weigh accurately about 1 g of sample, dissolve in 50 ml of water, add 50.0 ml of 0.05 *M* disodium EDTA and 20 ml of pH 4.5 buffer solution (77.1 g of ammonium acetate and 57 ml of glacial acetic acid in 1000 ml of solution), and boil gently for 5 min. Cool, and add 50 ml of alcohol and 2 ml of dithizone TS. Titrate with 0.05 *M* zinc sulfate to a bright rose-pink color, and perform a blank determination (see page 2). Each ml of 0.05 *M* disodium EDTA is equivalent to 22.67 mg of $AlNH_4(SO_4)_2 \cdot 12H_2O$.

Aluminum Potassium Sulfate, page 15

Replace the *Test* entitled *Assay* with the following (note that the buffer is added prior to the boiling step):

Assay Weigh accurately about 1 g of sample, dissolve in 50 ml of water, add 50.0 ml of 0.05 *M* disodium EDTA and 20 ml of pH 4.5 buffer solution (77.1 g of ammonium acetate and 57 ml of glacial acetic acid in 1000 ml of solution), and boil gently for 5 min. Cool, and add 50 ml of alcohol and 2 ml of dithizone TS. Titrate with 0.05 *M* zinc sulfate to a bright rose-pink color, and perform a blank determination (see page 2). Each ml of 0.05 *M* disodium EDTA is equivalent to 23.72 mg of $AlK(SO_4)_2 \cdot 12H_2O$.

Aluminum Sodium Sulfate, page 16

Replace the *Test* entitled *Assay* with the following (note that the buffer is added prior to the boiling step):

Assay Weigh accurately about 500 mg of sample previously dried as directed in the test for *Loss on Drying*, moisten with 1 ml of acetic acid, and dissolve in 50 ml of water, warming gently on a steam bath until solution is complete. Cool, neutralize with ammonia TS, add 50.0 ml of 0.05 *M* disodium EDTA and 20 ml of pH 4.5 buffer solution (77.1 g of ammonium acetate and 57 ml of glacial acetic acid in 1000 ml of solution), and boil gently for 5 min. Cool, and add 50 ml of alcohol and 2 ml of dithizone TS. Titrate with 0.05 *M* zinc sulfate to a bright rose-pink color, and perform a blank determination (see page 2). Each ml of 0.05 *M* disodium EDTA is equivalent to 12.10 mg of $AlNa(SO_4)_2$.

Aluminum Sulfate, page 17

Replace the *Test* entitled *Assay* with the following (note that the buffer is added prior to the boiling step):

Assay Weigh accurately an amount of sample equivalent to about 4 g of $Al_2(SO_4)_3$, transfer to a 250-ml volumetric flask, dissolve in water, dilute to volume, and mix. Pipet 10 ml of this solution into a 250-ml beaker, add 25.0 ml of 0.05 *M* disodium EDTA and 20 ml of pH 4.5 buffer solution (77.1 g of ammonium acetate and 57 ml of glacial acetic acid in 1000 ml of solution), and boil gently for 5 min. Cool, and add 50 ml of alcohol and 2 ml of dithizone TS. Titrate with 0.05 *M* zinc sulfate to a bright rose-pink color, and perform a blank determination (see page 2). Each ml of 0.05 *M* disodium EDTA is equivalent to 8.554 mg of $Al_2(SO_4)_3$ or to 16.66 mg of $Al_2(SO_4)_3 \cdot 18H_2O$.

Ammonium Bicarbonate, page 19

Replace the *Test* entitled *Assay* with the following:

Assay Weigh accurately about 3 g of sample, and dissolve it in 40 ml of water. Add 2 drops of methyl red TS, and titrate with 1 *N* hydrochloric acid. Add the acid slowly, with constant stirring, until the solution becomes faintly pink. Heat the solution to boiling, cool, and continue the titration until the faint pink color no longer fades after boiling. Each ml of 1 *N* hydrochloric acid is equivalent to 79.06 mg of NH_4HCO_3.

Aspartame, page 28

Change the *Description* to read:

DESCRIPTION

A white, odorless, crystalline powder having a sweet taste. It is sparingly soluble in water and slightly soluble in alcohol. The pH of a 0.8% aqueous solution is between 4.5 and 6.0.

Add to the *Requirement* entitled *Identification* a paragraph C to read:

C. Identify aspartame by comparing its infrared absorption spectrum with the spectrum shown on page 30, THIS SUPPLEMENT. The spectrum is obtained from a sample pelleted with KBr.

Change the *Requirement* entitled *Specific Rotation* to read:

Specific Rotation $[\alpha]_D^{20°}$ Between +14.5° and +16.5°, calculated on the dried basis.

Insert the following new monograph to precede the monograph entitled *Azodicarbonamide*, page 31.

Autolyzed Yeast Extract

DESCRIPTION

Autolyzed yeast extracts are composed primarily of (a) amino acids, peptides, and salts resulting from the acid-catalyzed hydrolysis of polypeptide bonds in naturally occurring enzymes present in the edible yeast and (b) the water-soluble components of the yeast cell. Food-grade salt may be added during processing. Individual products may be in liquid, paste, powder, or granular form. The pH of a 2% solution in water is between 4.5 and 6.0.

REQUIREMENTS

Calculate all determinations on the dried basis. Liquid and paste samples should be evaporated to dryness on a steam bath, then, as for the powdered and granular forms, dried to constant weight at 105° (see *General Provisions Applying to Specifications, Tests, and Assays of the Food Chemicals Codex*, page 1).

Assay (Total Nitrogen) Not less than 9.0% total nitrogen.
α-Amino Nitrogen Not less than 3.5%.
Arsenic Not more than 3 ppm.

Aspartic Acid Not more than 8.0% as $C_4H_7NO_4$ and not more than 12.0% of the total amino acids.
Glutamic Acid Not more than 12.0% as $C_5H_9NO_4$ and not more than 20.0% of the total amino acids.
Heavy Metals (as Pb) Not more than 0.002%.
Insoluble Matter Not more than 1%.
Lead Not more than 10 ppm.
Sodium Not more than 20.0%.

TESTS

Assay (Total Nitrogen) Proceed as directed under *Nitrogen Determination*, page 521.
α-Amino Nitrogen Proceed as directed in the test for *α-Amino Nitrogen* under *Acid Hydrolyzed Proteins*, page 3, THIS SUPPLEMENT.
Arsenic A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Arsenic Test*, page 464.
Aspartic and Glutamic Acids Proceed as directed in the test for *Aspartic and Glutamic Acids* under *Acid Hydrolyzed Proteins*, page 3, THIS SUPPLEMENT.
Heavy Metals Prepare and test a 1-g sample as directed in *Method II* under the *Heavy Metals Test*, page 513, using 20 μg of lead ion (Pb) in the control (*Solution A*).
Insoluble Matter Transfer about 5 g, accurately weighed, into a 250-ml Erlenmeyer flask, add 75 ml of water, cover the flask with a watch glass, and boil gently for 2 min. Filter the solution through a tared filtering crucible, dry at 105° for 1 h, cool, and weigh.
Lead A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Lead Limit Test*, page 518, using 10 μg of lead ion (Pb) in the control.
Sodium Proceed as directed in the test for *Sodium* under *Acid Hydrolyzed Proteins*, page 3, THIS SUPPLEMENT.

Packaging and Storage Store in well-closed containers.
Functional Use in Foods Flavoring agent.

Bay Oil, page 33

Delete the *Requirement* and *Test* for *Solubility in Alcohol*.

Calcium Oxide, page 55

Change the *Requirement* for *Fluoride* to read:

Fluoride Not more than 0.015%.

Carbon, Activated, page 70

Delete *Activated Charcoal* as a synonym.

Carmine, page 72

Replace the second sentence of the description with the following:

Cochineal consists of the dried female insects *Dactylopius coccus costa* (*Coccus cacti* L.) enclosing young larvae; the coloring principles derived therefrom consist chiefly of carminic acid ($C_{22}H_{20}O_{13}$).

Celery Seed Oil, page 78

Change the *Requirement* entitled *Acid Value* to read:

Acid Value Not more than 4.5.

Change the *Requirement* entitled *Saponification Value* to read:

Saponification Value Between 25 and 65.

Change the *Requirement* entitled *Specific Gravity* to read:

Specific Gravity Between 0.870 and 0.910.

Disodium Guanylate, page 105

Insert the following *Test* for *Loss on Drying*:

Loss on Drying, page 518 Dry at 120° for 4 h.

Enzyme Preparations, page 107

Under *Coliforms*, page 110, change Section 46.039 to Section 46.016.

Fructose, page 130

In the *Test* entitled *Assay*, change the factor for the 200-mm tube from 0.562 to 0.555 in the last sentence of the paragraph.

Insert the following new monograph to precede the monograph entitled *Guar Gum*, page 141:

Grape Skin Extract

Enocianina

DESCRIPTION

Grape skin extract is a red to purple powder or liquid concentrate prepared by aqueous extraction of grape marc remaining from the pressing of grapes to obtain juice. Extraction is effected with water containing sulfur dioxide. After concentration by vacuum evaporation, the sugar content is reduced by fermentation: further concentration removes most of the alcohol. The primary color components are anthocyanins such as the glucosides of malvidin, peonidin, petunidin, delphinidin, or cyanidin. Other components naturally present are sugars, tartrates, malates, tannins, and minerals. Alcohol or sulfur dioxide may be added. The powder may contain an added carrier such as maltodextrin, modified starch, or gum. In acid solution, grape skin extract is red; in neutral to alkaline solution, it is violet to blue.

REQUIREMENTS

Identification

Transfer 1 g of sample and 1 g of potassium metabisulfite to a 100-ml volumetric flask, dissolve in about 50 ml of pH 3.0 *Citrate-Citric Acid Buffer* (see *Assay* below), and dilute to volume with the same buffer. The red color due to anthocyanins is bleached.

Assay The color strength (*CS*) expressed as the absorbance of a 1% solution in a cell of 1-cm pathlength at pH 3.0 shall not be less than 90% of the color strength as represented.

Arsenic Not more than 3 ppm.

Lead Not more than 10 ppm.

Pesticides Pesticide levels shall conform with national regulations in the country of use.

Sulfur Dioxide Not more than 0.1%.

TESTS

Assay Transfer about 0.2 g of grape skin extract, accurately weighed, to a 100-ml volumetric flask, dissolve in about 25 ml of pH 3.0 *Citrate-Citric Acid Buffer*, and dilute to volume with the same buffer. (Prepare the buffer by adding 0.1 *M* sodium citrate dropwise to 0.1 *M* citric acid until a pH of 3.0 is reached, as determined by a glass electrode.) Allow this solution to stand in the dark for one-half hour, then remove any undissolved material by filtration or centrifugation. Adjust the pH to 3.0, and determine the absorbance of the clarified solution at 525 nm in a cell with a 1-cm pathlength. The color strength expressed as the absorbance of a 1% solution in a cell of 1-cm pathlength is calculated as:

$$CS = \text{Absorbance at 525 nm/Sample Weight in g.}$$

Arsenic A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Arsenic Test*, page 464.

Lead A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Lead Limit Test*, page 518, using 10 µg of lead ion (Pb) in the control.

Sulfur Dioxide Determine as directed in the general method, page 546. Omit preparation of the slurry in 300 ml of recently boiled and cooled water. Transfer the sample directly to the flask, dilute to 400 ml with water, and proceed as directed.

Packaging and Storage Store liquid grape skin extract in high-density polyethylene containers at 4°–14°. Store powdered grape skin extract in fiber drums at room temperature.

Functional Use in Foods Color.

L-Isoleucine, page 154

Change the *Molecular Weight* from 131.18 to 131.17.

L-Leucine, page 171

Change the *Molecular Weight* from 131.18 to 131.17.

Magnesium Oxide, page 178

Change the *Requirement* entitled *Heavy Metals (as Pb)* to read:

Heavy Metals (as Pb) Not more than 0.004%.

Insert the following revised sections for *Magnesium Sulfate* immediately preceding and following the *Tests*, which are unchanged:

Magnesium Sulfate, page 183

Epsom Salt

$MgSO_4 \cdot xH_2O$ Mol wt (anhydrous) 120.36

DESCRIPTION

Magnesium sulfate is produced with one or seven molecules of water of hydration, or in a dried form containing the equivalent of about 2.3 waters of hydration. It is in the form of a colorless crystal or a granular crystalline powder. It is odorless. It is readily soluble in water, slowly soluble in glycerine, and sparingly soluble in alcohol.

REQUIREMENTS

Identification

A 1 in 20 solution gives positive tests for *Magnesium*, page 517, and for *Sulfate*, page 517.

Assay Not less than 99.5% of $MgSO_4$ after ignition.
Arsenic (as As) Not more than 3 ppm.
Heavy Metals (as Pb) Not more than 10 ppm.
Loss on Ignition Between 13% and 16% for the monohydrate. Between 22% and 28% for the dried form. Between 40% and 52% for the heptahydrate.
Selenium Not more than 0.003%.

Packaging and Storage Store in well-closed containers.
Labeling Label magnesium sulfate to indicate whether it is the monohydrate, the dried form, or the heptahydrate.
Functional Use in Foods Nutrient; dietary supplement.

Mandarin Oil, Coldpressed, page 185

Change the *Requirement* entitled *Specific Gravity* to read:

Specific Gravity Between 0.846 and 0.852.

Peppermint Oil, page 219

In the *Test* entitled *Assay for Total Menthol*, change the formula for calculating the percentage of total menthol from

$$7.814A(0.0021E)/(B - 0.021A)$$

to

$$7.814A(1 - 0.0021E)/(B - 0.021A).$$

Petroleum Wax, Synthetic, page 222

In the paragraph entitled *Ultraviolet Absorbance*, page 223, change the CFR reference from 21 CFR 121.1156 to 21 CFR 172.886.

Potassium Alginate, page 239

Change the *Requirement* entitled *Assay* to read:

Assay It yields not less than 16.5% and not more than 19.5% of carbon dioxide (CO_2), corresponding to between 89.2% and 105.5% of potassium alginate (equiv wt 238.00), calculated on the dried basis.

Change the *Requirement* entitled *Heavy Metals* to read:

Heavy Metals (as Pb) Not more than 0.004%.

Insert the following new *Requirement* to precede the *Requirement* entitled *Loss on Drying*:

Lead Not more than 10 ppm.

Insert the following *Test* entitled *Lead* immediately preceding *Loss on Drying*:

Lead A *Sample Solution* prepared as directed for organic compounds meets the requirements of the *Lead Limit Test*, page 518, using 10 µg lead ion (Pb) in the control.

Potassium Bicarbonate, page 239

Replace the *Test* entitled *Assay* with the following:

Assay Weigh accurately about 4 g of sample, and dissolve it in 100 ml of water. Add 2 drops of methyl red TS, and titrate with 1 *N* hydrochloric acid. Add the acid slowly, with constant stirring, until the solution becomes faintly pink. Heat the solution to boiling, cool, and continue the titration until the pink color no longer fades after boiling. Each ml of 1 *N* hydrochloric acid is equivalent to 100.1 mg of $KHCO_3$.

Potassium Carbonate, page 240

Replace the *Test* entitled *Assay* with the following:

Assay Weigh accurately in a stoppered weighing bottle about 1 g of the dried sample obtained as directed under *Loss on Drying*, and dissolve it in 50 ml of water. Add 2 drops of methyl red TS, and titrate with 1 *N* hydrochloric acid. Add the acid slowly, with constant stirring, until the solution becomes faintly pink. Heat the solution to boiling, cool, and continue the titration until the faint pink color no longer fades after boiling. Each ml of 1 *N* hydrochloric acid is equivalent to 69.10 mg of K_2CO_3.

Potassium Nitrate, page 247

Change the *Requirement* entitled *Identification* to read:

Identification

A 1 in 10 solution gives positive tests for *Potassium*, page 517, and *Nitrate*, page 517.

Potassium Sorbate, page 252

Change the structure shown from $CH_3CH=CHCH=COOK$ to $CH_3CH=CHCH=CHCOOK$.

L-Serine, page 270

Change the *Molecular Weight* from 105.10 to 105.09.

Silicon Dioxide, page 271

Delete the *Requirement* entitled *Insoluble Substances*, page 272.

Sodium Alginate, page 274

Replace the last sentence of the *Requirement* entitled *Assay* with the following:

Each ml of 0.25 *N* sodium hydroxide consumed in the assay is equivalent to 27.75 mg of sodium alginate (equiv wt 222.00), calculated on the dried basis.

Sodium Bicarbonate, page 278

Replace the *Test* entitled *Assay* with the following:

Assay Weigh accurately about 3 g of sample, previously dried over silica gel for 4 h, and dissolve it in 100 ml of water. Add 2 drops of methyl red TS, and titrate with 1 *N* hydrochloric acid. Add the acid slowly, with constant stirring, until the solution becomes faintly pink. Heat the solution to boiling, cool, and continue the titration until the faint pink color no longer fades after boiling. Each ml of 1 *N* hydrochloric acid is equivalent to 84.01 mg of $NaHCO_3$.

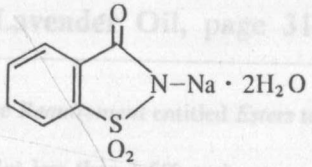
Sodium Carbonate, page 280

Replace the *Test* entitled *Assay* with the following:

Assay Weigh accurately about 2 g of the dried salt, obtained as directed under *Loss on Drying*, and dissolve it in 50 ml of water. Add 2 drops of methyl red TS, and titrate with 1 *N* hydrochloric acid. Add the acid slowly, with constant stirring, until the solution becomes faintly pink. Heat the solution to boiling, cool, and continue the titration until the faint pink color no longer fades after boiling. Each ml of 1 *N* hydrochloric acid is equivalent to 53.00 mg of Na_2CO_3.

Sodium Saccharin, page 297

Replace the molecular structure with the following:



Spice Oleoresins, page 310

Insert the following new *Spice Oleoresins* in the proper alphabetical order:

Oleoresin Angelica Seed Obtained by the solvent extraction of the dried seed of *Angelica archangelica* L. as a dark brown or green liquid.

Oleoresin Anise Obtained by the solvent extraction of the dried ripe fruit of *Pimpinella anisum* L. or *Illicium verum* L. as a dark brown or green liquid.

Oleoresin Basil Obtained by the solvent extraction of the dried plant of *Ocimum basilicum* L. as a dark brown or green semisolid.

Oleoresin Caraway Obtained by the solvent extraction of the dried seeds of *Carum carvi* L. as a green yellow to brown liquid.

Oleoresin Cardamom Obtained by the solvent extraction of the dried seeds of *Elettaria cardamomum Maton* as a dark brown or green liquid.

Oleoresin Coriander Obtained by the solvent extraction of the dried seeds of *Coriandrum sativum* L. as a brown yellow to green liquid.

Oleoresin Cubeb Obtained by the solvent extraction of the dried fruit of *Piper cubeba* L. as a green or green brown liquid.

Oleoresin Cumin Obtained by the solvent extraction of the dried seeds of *Cuminum cyminum* L. as a brown to yellow green liquid.

Oleoresin Dillseed Obtained by the solvent extraction of the dried seeds of *Anethum graveolens* L. as a brown or green liquid.

Oleoresin Fennel Obtained by the solvent extraction of the dried fruit of *Foeniculum vulgare Miller* as a brown green liquid.

Oleoresin Laurel Leaf Obtained by the solvent extraction of the dried leaves of *Laurus nobilis* L. as a dark brown or green semisolid.

Oleoresin Marjoram Obtained by the solvent extraction of the dried herb of the marjoram shrub *Majorama hortensis Moench* as a dark green to brown viscous liquid or semisolid.

Oleoresin Origanum Obtained by the solvent extraction of the dried flowering herb *Origanum vulgare* L. as a dark brown green semisolid.

Oleoresin Parsley Leaf Obtained by the solvent extraction of the dried herb of *Petroselinum crispum* L. as a brown to green liquid.

Oleoresin Parsley Seed Obtained by the solvent extraction of the dried seeds of *Petroselinum crispum* L. as a deep green semiviscous liquid.

Oleoresin Pimenta Berries Obtained by the solvent extraction of the dried fruit of *Pimenta officinalis Lindley* as a brown green to dark green liquid.

Oleoresin Thyme Obtained by the solvent extraction of the dried flowering plant *Thymus vulgaris* L. as a dark brown to green, viscous semisolid.

Insert the following in the proper alphabetical order under *Additional Requirements*, page 311:

Oleoresin Angelica Seed *Volatile Oil Content:* between 2 ml and 7 ml per 100 g.
Oleoresin Anise *Volatile Oil Content:* between 9 ml and 22 ml per 100 g.
Oleoresin Basil *Volatile Oil Content:* between 4 ml and 17 ml per 100 g.
Oleoresin Caraway *Volatile Oil Content:* between 10 ml and 20 ml per 100 g.
Oleoresin Cardamom *Volatile Oil Content:* between 50 ml and 80 ml per 100 g.
Oleoresin Coriander *Volatile Oil Content:* between 2 ml and 12 ml per 100 g.
Oleoresin Cubeb *Volatile Oil Content:* between 50 ml and 80 ml per 100 g.
Oleoresin Cumin *Volatile Oil Content:* between 10 ml and 30 ml per 100 g.
Oleoresin Dillseed *Volatile Oil Content:* between 10 ml and 20 ml per 100 g.
Oleoresin Fennel *Volatile Oil Content:* between 3 ml and 20 ml per 100 g.
Oleoresin Laurel Leaf *Volatile Oil Content:* between 5 ml and 25 ml per 100 g.
Oleoresin Marjoram *Volatile Oil Content:* between 10 ml and 20 ml per 100 g.
Oleoresin Origanum *Volatile Oil Content:* between 20 ml and 45 ml per 100 g.
Oleoresin Parsley Leaf *Volatile Oil Content:* between 2 ml and 10 ml per 100 g.
Oleoresin Parsley Seed *Volatile Oil Content:* between 2 ml and 7 ml per 100 g.
Oleoresin Pimenta Berries *Volatile Oil Content:* between 20 ml and 50 ml per 100 g.
Oleoresin Thyme *Volatile Oil Content:* between 5 ml and 12 ml per 100 g.

Spike Lavender Oil, page 311

Change the *Requirement* entitled *Esters* to read:

Esters Not less than 1.5% and not more than 4.0% of esters calculated as linalyl acetate ($C_{12}H_{20}O_2$).

d-α-Tocopheryl Acetate Concentrate, page 335

Change the *Requirement* entitled *Identification* to read:

Identification

It meets the requirements of *Identification Tests A* and *B* under *d*-α-*Tocopheryl Acetate*, page 333.

Change the *Test* entitled *Assay* to read:

Assay Proceed as directed in the *Assay* under *d-α-Tocopheryl Acetate*, page 333, using the following as the *Assay Preparation*: Dissolve an accurately weighed amount of the sample equivalent to about 30 mg of *d-α*-tocopheryl acetate in 10.0 ml of the *Internal Standard Solution*.

Triacetin, page 337

Replace the sentence describing *Packaging and Storage*, page 338, with the following:

Packaging and Storage Store in well-closed containers.

Triethyl Citrate, page 339

In the *Test* entitled *Assay*, replace phenolphthalein with bromothymol blue in the fourth line from the bottom of the paragraph.

Xylitol, page 349

In the *Test* entitled *Other Polyols*, in the paragraph labeled *Procedure*, change the approximate retention time for mannitol hexaacetate from 30 min to 20 min.

Zinc Sulfate, page 351

Change the *Requirement* entitled *Assay*, for the monohydrate only, to read:

Assay *Monohydrate:* not less than 98.0% and not more than 100.5% of $ZnSO_4 \cdot H_2O$.

Change the last sentence of the *Test* entitled *Assay* to read:

Each ml of 0.05 *M* disodium EDTA is equivalent to 8.973 mg of $ZnSO_4 \cdot H_2O$ or 14.38 mg of $ZnSO_4 \cdot 7H_2O$.